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Dielectric properties of starch slurries as influenced by starch concentration and gelatinization

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Abstract

Dielectric properties of corn starch slurries of 10–50% (w/w) starch concentration were measured at temperatures between 40 and 90 °C and in the frequency range from 15 MHz to 3 GHz. Dielectric constant decreased and the loss factor increased with increasing starch concentrations. The dielectric loss factor of the starch slurries decreased at frequencies 15–450 MHz and increased at frequencies 450 MHz to 3 GHz. A significant effect of frequency on the dielectric constant was evident only at higher starch concentrations (>40%). Dielectric constant of starch slurries decreased with increasing temperature throughout the frequency range while the dielectric loss increased with temperature between 15 MHz and 450 MHz and then decreased with increase in temperature between 450 MHz and 3 GHz. During gelatinization, the dielectric constant for 20% starch slurry decreased in a linear fashion. A significant correlation ($R^2 = 0.87611$; $P < 0.05$) was observed between the dielectric constant and the percentage gelatinization for the 20% starch slurry in the temperature range from 60 to 72 °C.

Keywords: Dielectric constant; Dielectric loss factor; Frequency; Gelatinization

1. Introduction

Dielectric materials are characterized by their ability to support electric fields and not conduct electric current (Shivola, 1999). The ability of these materials to store and dissipate the electric energy is described by their dielectric properties. These properties are the key factors to understanding the interactions of dielectric materials with microwave or radio frequency fields. The relevant dielectric properties of food materials are the dielectric constant and the dielectric loss factors that are real and imaginary parts, respectively, of the relative complex permittivity (ε^*) expressed as

$$\varepsilon^* = \varepsilon' - i\varepsilon'' \tag{1}$$

Dielectric constant (ε') is indicative of the ability of the material to store energy and polarize when subjected to an electric field, while the dielectric loss factor (ε'') is associated with loss of electric filed energy in material which is dissipated as heat (Decareau, 1985; Nelson, 1973).

In food systems, water is the major polar material and hence, amount and mobility of water influence the dielectric properties of the system. A change in the water content of the system changes the amount of dipolar material interacting with the electromagnetic waves, thus affecting the dielectric properties. Furthermore, the presence of water causes certain physico-chemical changes in the system such as changes in viscosity, starch gelatinization on heating starch granules with water and glass transition changes. These transitions also involve changes in the mobility of water, thereby affecting the dielectric properties of the system (Miller, Gordon, & Davis, 1991; Tsoubelli, Davis, & Gordon, 1995).

Starches are widely used in foods as thickening agents due to their swelling and solubilization properties at and above gelatinization temperatures (Self, Wilkin, Morley, & Bailey, 1990). Starch gelatinization is characterized by

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disruption of its native granular structure accompanied by swelling of granules, leaching of amylose, loss of crystallinity, loss of birefringence, increase in viscosity and solubilization of starch polymers (Atwell, Hood, Lineback, Varriano-Martson, & Zobel, 1988). Granule swelling during gelatinization results in an increase in viscosity with a corresponding decrease in water mobility. The average mobility depends on the ratio of bound water molecules to the total moisture content of the system and on the mobility of starch molecules. The decrease in the mobility will have an impact on the dielectric properties of the starch–water systems (Jaska, 1971; Ryynänen, Risman, & Ohlsson, 1996).

Several researchers have investigated the influence of water content, starch gelatinization and temperature on the dielectric properties of different starches employing various techniques of measurement (Bircan & Barringer, 1998; Ndife, Şumnu & Bayindirli, 1998a, 1998b; Piyasena, Ramaswamy, Awuah, & Defelice, 2003; Ryynänen et al., 1996). Dielectric constant and loss factor of starches depend on moisture content, temperature and type of starch. Dielectric properties of dry starch granules equilibrated to different water activities were considerably lower than those of starch–water systems with 33–50% (w/w) starch concentrations, when measured with a dielectric probe. Generally, with increase in water content, the dielectric constant and the loss factor have been reported to increase (Ndife et al., 1998a; Tsoubelli et al., 1995). However, Bircan and Barringer (1998) reported that at a constant temperature of 25 °C, with increase in water content of the system from 75% to 100% (w/w), the dielectric loss factor remained unchanged, though the dielectric constant increased. Dielectric properties of starch–water systems decreased following heating when measured at 2450 MHz (Ndife et al., 1998a).

When measurements were conducted by cavity perturbation technique at 2750 MHz for corn, wheat, potato and waxy corn starch in excess water systems (5–30% starch), the differences in dielectric properties due to starch type and starch gelatinization were not significant, though the dielectric properties decreased with temperature and concentration of starch (Ryynänen et al., 1996). Although the reported dielectric properties by various independent studies differed, it is generally concluded that dielectric properties depend primarily on free water content. The effect of gelatinization on the dielectric properties of starch has not been adequately explained and the changes in dielectric properties during gelatinization have not been reported in detail.

Starch gelatinization has been studied by observing the various physico-chemical changes occurring in starch granules during heating in aqueous systems. Some of the most common methods include the observation of granule swelling and loss of birefringence by optical microscopy (Atwell et al., 1988), loss of crystallinity as observed by X-ray diffraction (Zobel, Young, & Rocca, 1988), thermal changes as observed by differential scanning calorimetry (Biliaderis, Maurice, & Vose, 1980) and changes in electrical resistivity (Leszczynski, 1987). Abrupt changes in dielectric properties of settled and suspended potato starches were observed near gelatinization temperatures, demonstrating that dielectric properties can be used to observe starch gelatinization (Sipahioglu, Bircan, & Barringer, 2004). Dielectric-dissipation-induced changes have also been employed to determine the gelatinization onset and end points and energy of transformation. The curves of dissipative alternating current vs. temperature were reported to exhibit a peak when starch starts absorbing water (Morales-Sánchez et al., 1997). Also, differential dissipation factor (DDF) has been used for measuring structural changes in starch-derived foods during processing. In the frequency range of 1–25 Hz, the DDF has a peak whose maximum depends on the threshold temperature; i.e., temperature above and below which different behaviour patterns for the various properties are observed. This peak was related to the changes in bound water, as it is sensitive to the structural changes and therefore to the gelatinization of the starch samples (Aŕambula et al., 1998).

The objective of this research was to determine the impact of temperature and frequency of measurement, and starch gelatinization on the dielectric properties of starch–water systems at various starch concentrations and to develop a technique to determine the degree of starch gelatinization by measuring dielectric properties.

2. Materials and methods

2.1. Preparation of starch slurries

Slurries of corn starch (Argo corn starch, Memphis, TN) at 10%, 20%, 30%, 40% or 50% (w/w) concentrations were prepared by adding deionised water at room temperature. The slurry was stirred on a magnetic stirrer for 30 min. Samples were deaerated by aspirating for 15 min to remove any air bubbles. The samples were then heated to six end different temperatures between 40 and 90 °C at intervals of 10 °C for all starch concentrations Additional measurements were also conducted at 3 °C intervals between 60 and 75 °C for the 20% and 50% starch slurries. The slurries were heated in a air convection oven (Fisher Scientific, Isotemp Oven Model 630 F). Following heating to specific end temperatures, the dielectric properties of the slurries were measured. Each experiment was conducted in triplicate and each sample was used for just one end temperature.

2.2. Dielectric measurements

The dielectric properties were measured by using a network analyzer (HP 8527 C, Hewlett Packard Co., Santa Rosa, CA) with an open ended coaxial line connected to a dielectric probe (HP 85070B, Hewlett Packard Co., Santa Rosa, CA) in a frequency range of 500–3000 MHz. The network analyzer was calibrated with air, metallic short and water at 25 °C and then recalibrated with water at the respective temperature of measurement just before the measurement was made to ensure the accuracy of measurement. This also prevented the sample to cool down to a lower temperature when it came in contact with the dielectric probe. Samples were placed in a glass beaker encased with a surrounding Styrofoam stand to minimize the heat loss to surroundings. The reflection measurement technique was used because of its relative ease of use at higher temperatures. The dielectric properties were calculated using the HP software HP 85070 using the scattering parameter S_{11}.

2.3. Measurement of extent of gelatinization

The thermal properties of the 20% and 50% starch slurries were analyzed with a differential scanning calorimeter, DSC-7 equipped with a Pyris software for Windows package, version 2.04, 1997 (Perkin-Elmer Corp., Norwalk, CT, USA). Slurries were weighed in stainless steel (SS) pan and sealed. An empty SS pan was used as a reference in the DSC. Samples were held at 20 °C for 5 min and then heated from 20 to 140 °C at a heating rate of 1.5 °C/min. This heating rate was chosen to simulate the heating rate of samples heated in the convection oven used for dielectric measurements. Gelatinization onset and end temperatures as well as gelatinization enthalpy were calculated from the endothermic peak by using the DSC software. The enthalpy of the completely gelatinized starch was termed as enthalpy of complete gelatinization (ΔH_c).

In subsequent DSC experiments, samples were held at 60, 63, 64, 66, 68, 70 or 72 °C for 5 min and the gelatinization enthalpy was obtained. This enthalpy was termed as enthalpy of partial gelatinization (ΔH_p). The degree of gelatinization was obtained by using the following equation (Ndife, Şumnu, & Bayindirli, 1998b);

$$\%\ \text{Gelatinization} = \left(1 - \frac{\Delta H_p}{\Delta H_c}\right)100 \qquad (2)$$

2.4. Statistical analysis

All measurements were conducted at least in triplicate. A crossed design with starch concentration (10%, 15%, 20%, 30%, 40% and 50%) and temperature (40, 50, 60, 70, 80 and 90 °C) as fixed factors and 4 frequencies (915, 1800, 2450 and 3000 MHz) as the random factors was used for analysis of variance at 0.05% level of significance using the general linear model of Minitab software (Minitab Inc., State College, PA, ver.14.0).

3. Results and discussion

The dielectric properties of corn starch slurries were measured as a function of starch concentration, frequency and the temperature.

3.1. Effect of temperature and frequency of measurement

The dielectric constant of starch slurries decreased significantly ($p < 0.05$) with increasing temperature (Fig. 1). The magnitude of this decrease varied with the temperature range and a relatively large decrease was observed between 50 and 60 °C, and between 60 and 70 °C, as compared to the decrease in the other temperature ranges. The dielectric loss factor exhibited an increase with the temperature in the frequency range of 15–450 MHz and a decrease with the temperature in the higher frequency range of 450 MHz–3 GHz. The increase in loss factor with temperature in the lower frequency range and the decrease in the higher frequency range was significant ($p < 0.001$) in the intermediate temperature ranges. The change in loss factor due to temperature was more extensive in the lower frequency range of 15–450 MHz. The dielectric behavior of starch slurries was similar to the behavior of pure water in the examined frequency range.

Temperature dependence of dielectric properties is usually explained by relaxation phenomena occurring at that temperature and frequency range. Normally the relaxation time of the polar molecules (time required for the dipoles to revert to random order when the electromagnetic field is

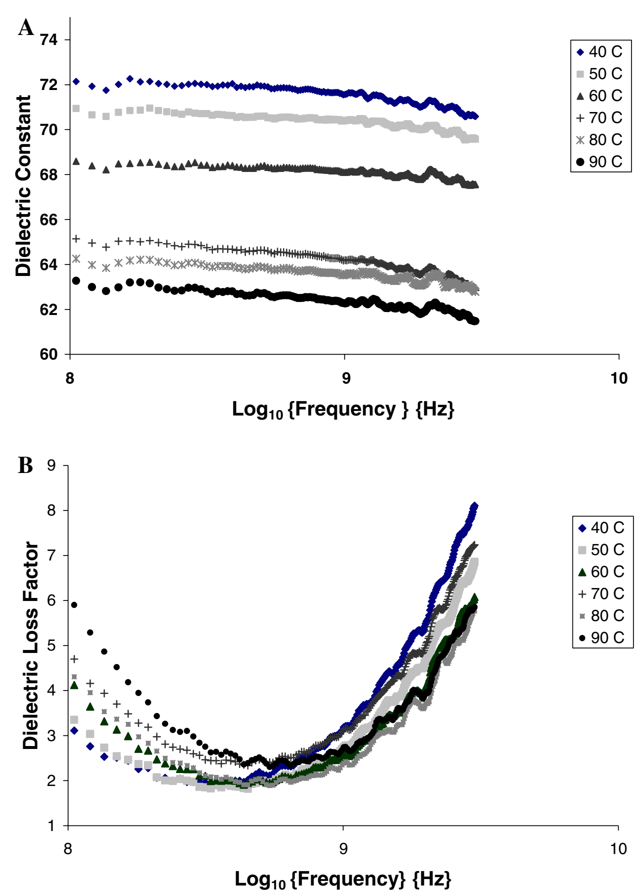

Fig. 1. Dielectric constant (A) and loss factor (B) of 15% corn starch slurry at six end temperatures and from 100 MHz to 3 GHz plotted on a logarithmic scale.

removed) decreases with increasing temperature and hence the dielectric constant should increase with increase in the temperature in the region of dispersion. In the absence of dielectric loss, the dielectric constant for such materials decreases with increasing temperature. The decrease in dielectric properties with temperature has been previously observed (Ndife et al., 1998a; Piyasena et al., 2003) and has been attributed to interactions between conductivity, rheological effects and relaxation times (Piyasena et al., 2003).

At all concentrations, the dielectric constant and loss factor decreased in the lower frequency range (15–450 MHz) and increased in the higher frequency range (450 MHz–3 GHz). With increase in frequency, the polar molecules are unable to keep up with change in the orientation of the imposed electromagnetic field, thus causing a lag. This results in dissipation of energy, which is indicated by decrease in dielectric constant and increase in the dielectric loss factor. Little or no change in dielectric constant at low concentration suggests the absence of dispersion in the frequency range measured. This behaviour of starch slurries is similar to the behaviour of pure water in the same frequency range. This result is in accordance with data reported by Piyasena et al. (2003) who reported similar trends in relative permittivity at radio frequencies.

3.2. Effect of starch concentration

The impact of starch concentration on the dielectric behaviour of starch slurries in the measured frequency range is shown in Fig. 2. The dielectric constant decreased with increasing starch concentration. At lower starch concentrations (<40%), the dielectric constant did not significantly change with frequency. However, at higher concentrations, it decreased with increasing frequency. On the other hand, the dielectric loss factor increased with increasing concentration of starch in the measured concentration range. The frequency behavior of dielectric loss at all concentrations exhibited a U-shaped curve which has been discussed above.

The impact of starch concentration and temperature on the dielectric constant and the loss factor at 2450 MHz is shown in Fig. 3. This frequency was chosen because it is the frequency at which domestic microwave ovens operate. The dielectric constant of starch slurries decreased significantly with increase in starch concentration in the range from 10% to 50% ($p < 0.05$) (Fig. 3). The observed decrease in the dielectric constant between 50–60 °C and 60–70 °C became more pronounced as starch concentration increased. Since this temperature range is near the gelatinization temperature range of starch, the observed behaviour could be attributed to the decrease in mobility of water due to gelatinization. Restricted mobility of water dipoles can restrict the ability of molecules to align with the alternating electric field. At lower concentrations (<40%), there was little change in the dielectric constant after 70 °C while for the 50% starch slurry, the dielectric constant increased.

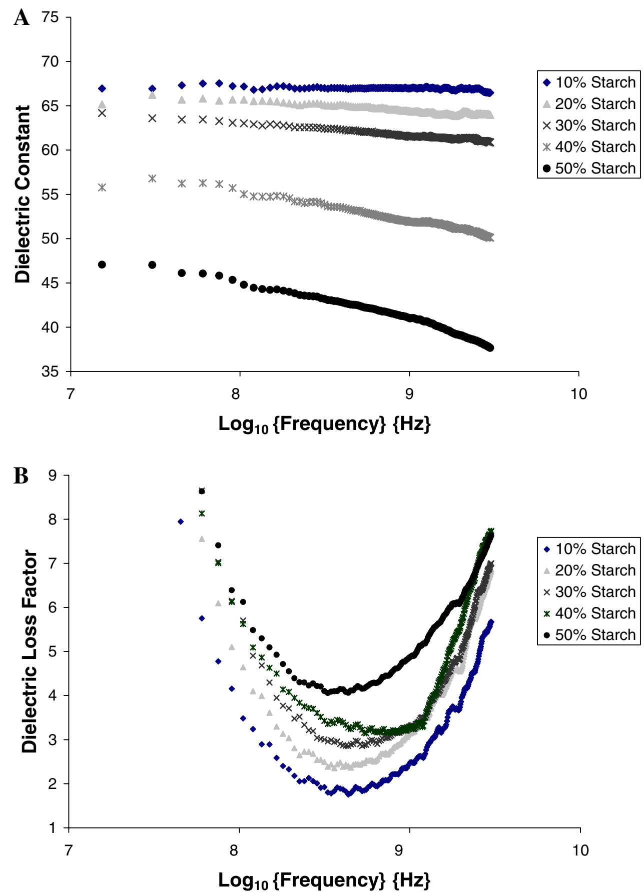

Fig. 2. Dielectric constant (A) and the loss factor (B) of corn starch slurries at 60 °C in the frequency range of 300 kHz–3 GHz, plotted on a logarithmic scale.

This behavior is contrary to the trend observed at lower starch concentrations. However, such anomaly is well recognized in the starch literature, wherein it is recognized that around a starch to water concentration of 1:1, i.e., limited moisture systems, the particular events associated with starch gelatinization, including granule swelling, amylopectin crystal melting, the uncoiling of the polymer helices and the amount of additional unfrozen water (i.e., bound water) are different (Farhat, Blanshard, & Mitchell, 2000; Keetels, van Vliet, & Walstra, 1996a, 1996b; Tananuwong & Reid, 2004; Waigh, Gidley, Komanshenk, & Donald, 2000). Table 1 also shows the effect of starch concentration on the gelatinization enthalpy as measured by using a DSC. The enthalpy of gelatinization decreased above a starch concentration of 30%. This decrease is related to molecular events in the granule that in the starch literature has been defined as the G-endotherm and the M1-endotherm. These two melting events separate or merge together based on starch concentration or the heating rate (Biliaderis, Page, Maurice, & Juliano, 1986; Donovan, 1979) and therefore influence the measured enthalpy. The observations reported in this manuscript are the first to demonstrate likely differences in the dielectric properties as a function of starch concentration in the gelatinization temperature range that

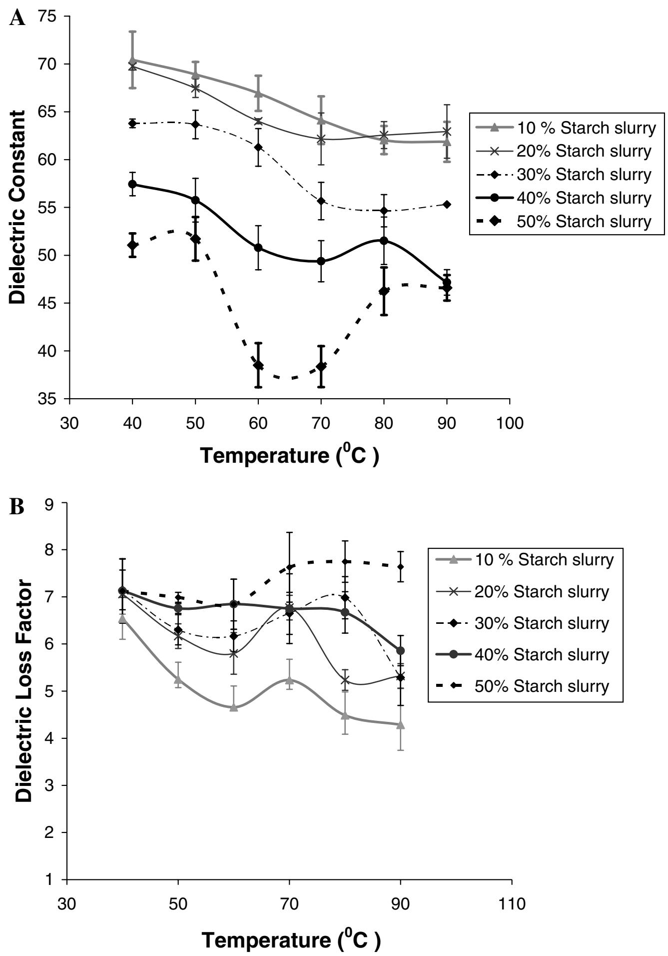

Fig. 3. Effect of starch concentration and temperature on the dielectric constant (A) and the loss factor (B) of corn starch slurries as measured at 2450 MHz.

Table 1
Gelatinization onset temperature (T_o), end temperature (T_e), and gelatinization enthalpy (ΔH) values of corn starch slurries at different starch concentrations

Starch slurry concentrations % (w/w)	Gelatinization onset temperature T_o (°C)	Gelatinization end temperature T_e (°C)	Gelatinization enthalpy ΔH (J/g)
10	65.37 ± 0.53	72.885 ± 0.70	9.91 ± 1.14
15	65.55 ± 0.12	72.87 ± 0.11	8.85 ± 0.04
20	65.29 ± 0.05	73.27 ± 0.24	9.314 ± 0.63
30	65.09 ± 0.1	73.68 ± 0.22	9.06 ± 0.65
40	64.25 ± 0.6	72.79 ± 0.09	4.90 ± 0.98
50	61.97 ± 2.62	72.34 ± 0.97	3.07 ± 1.10

might be indicative of or reflecting other molecular events occurring during gelatinization.

The dielectric loss factor increased with starch concentration, which indicates an increase in ability to dissipate the energy in the form of heat. Thus, starch concentration needs to be taken into consideration while developing microwaveable foods that use starch as a thickening agent (Piyasena et al., 2003). It was interesting to observe that while the dielectric constant showed a remarkable change near gelatinization, which was pronounced at relatively higher concentrations (>10%), the loss factor showed a peak near gelatinization at lower concentrations (<20%). The interaction of starch concentration and temperature on both the dielectric properties was statistically significant ($p < 0.001$).

The behavior of dielectric constant was found to depend on the water content, which agrees with results reported in the literature (Bircan & Barringer, 1998; Ndife et al., 1998a; Piyasena et al., 2003; Ryynänen et al., 1996). This has been attributed to change in the polarizable dipole moments per unit volume of the sample with change in free water content of the sample. However, the increase in amount of starch concentration (accompanied by a decrease in water content) has been reported to decrease dielectric loss (Butler & Cameron, 2000; Ndife et al., 1998a; Tsoubelli et al., 1995) which is opposite to what was observed in this study. It is likely that with an increase in the amount of starch in the system, the dielectric relaxation effects due to bound water in the molecular chains in the starch polymer start to interact with and possibly modify the dielectric influence of free water in the system.

3.3. Effect of starch gelatinization

The change in the trend of the dielectric properties of the concentrated starch water system shown in Fig. 3 prompted us to further investigate the changes in dielectric properties within the narrower gelatinization temperature range (60–72 °C). In this temperature range, the trends in dielectric properties differed with starch concentration. At 20% starch concentration, the dielectric constant value decreased almost linearly (Fig. 4), while at 50% starch concentration the dielectric constant appeared to peak at 66 °C and remained unchanged thereafter until 72 °C (Fig. 5). The results of dielectric loss factor in the gelatinization range confirmed the increase in the loss factor value during gelatinization. However at 50% starch concentration, the loss factor of the slurry did not show any significant variation with temperature.

Gelatinization produces a change in the mobility of water molecules in the system and hence has the ability to influence the dielectric properties. With increasing starch concentration, the changes in the dielectric constant of the system appeared more extensive and nonlinear. This is further confirmed by Ryynänen et al. (1996) wherein they reported little changes due to gelatinization on the dielectric properties of potato starch–water systems at low starch concentration and a drastic increase in the dielectric constant in granular starches equilibrated to a water activity of 0.6 at gelatinization temperature range.

3.4. Model to predict the extent of gelatinization of starch

The dielectric constant of the 20% starch slurry exhibited an almost linear decrease in the gelatinization temperature range (60–72 °C) (Fig. 4). The extent of gelatinization

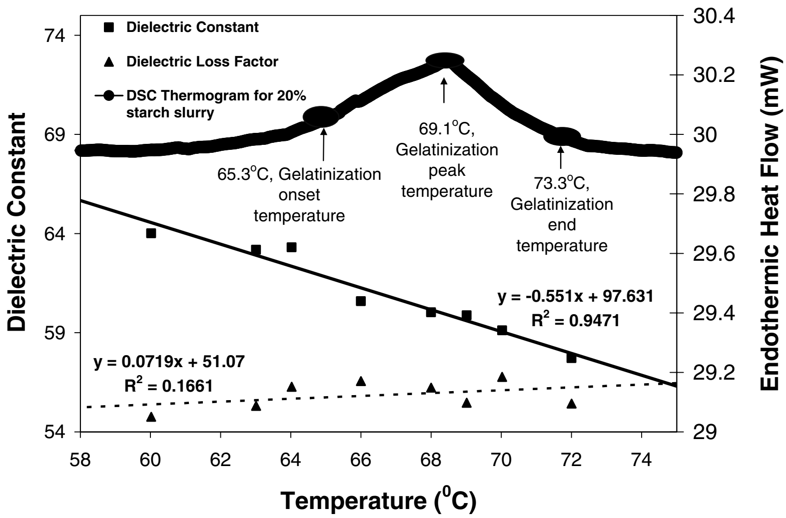

Fig. 4. Dielectric constant and loss factor of 20% corn starch slurry at 2450 MHz and DSC endotherm of 20% starch slurry. A value of 50 has been added to the dielectric loss factor data in order to fit into the same scale in the figure.

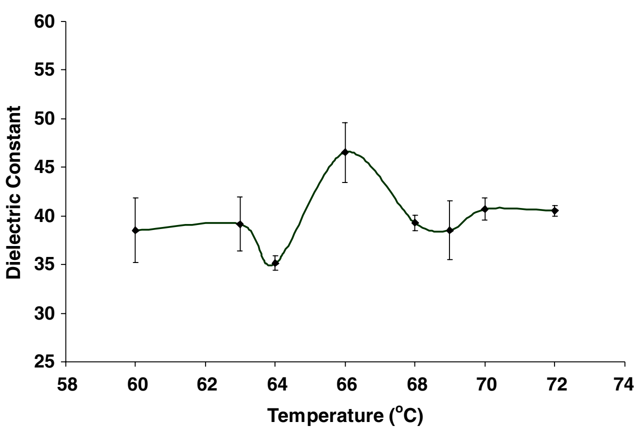

Fig. 5. Dielectric constant of 50% corn starch slurry at 2450 MHz during gelatinization.

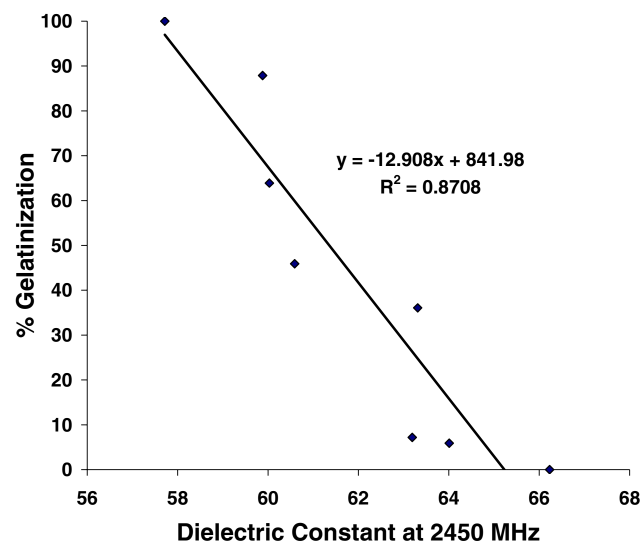

Fig. 6. % Gelatinization vs. dielectric constant at 2450 MHz for 20% starch slurry.

of starch at various temperatures in the gelatinization range was plotted by using data obtained from the DSC thermograms (Fig. 6). This was used as the basis for developing the model for predicting the gelatinization of starch by measuring the dielectric properties. The model ($p < 0.05$) is represented as

$$\% \text{ Gelatinization} = -12.908 \times \text{DC (at 2450 MHz)} + 841.98; \; R^2 = 0.8708 \quad (3)$$

where DC is the dielectric constant. Due to the nonlinear behavior of dielectric loss during gelatinization, the R^2 value obtained when it was correlated with extent of gelatinization was quite low (0.0741). The higher R^2 value obtained for the dielectric constant indicates that it is a better indicator for predicting the extent of starch gelatinization. This is expected because the dielectric constant is influenced by the orientational changes occurring in the starch–water system.

4. Conclusions

This study showed that dielectric properties of starch slurries are significantly influenced by temperature, starch gelatinization and starch concentration. Dielectric constant decreased when the temperature was increased, which was attributed to decrease in the relaxation time of the molecules in the system as the temperature increased. The effect of temperature on loss factor, however, was observed to be frequency dependent. The frequency of measurement had little effect on the dielectric constant of 10–30% starch slurries, while for 40% and 50% starch slurries, dielectric constant decreased with increase in frequency. The dielectric loss factor first decreased till 450 MHz and then increased, indicating a U-shaped curve behavior with the frequency. The dielectric constant decreased with the starch concentration, which could result from reduction in the polarizability of water molecules. However, the constant increased post-gelatinization at higher starch concentrations. The loss factor increased with concentration of starch, thus leading to the inference that heat dissipated in the starch–water system during microwave heating would increase with the starch concentration. In the gelatinization temperature range, the dielectric constant exhibited an almost linear decrease with temperature for 20% starch slurry and hence, yielded R^2 of 0.87 when correlated with the extent of gelatinization calculated by DSC melting endothermic peaks during starch gelatinization. The dielectric loss factor exhibited a non linear behavior with temperature and an abrupt increase was observed during gelatinization yielding a lower R^2 value for its correlation with the extent of gelatinization. Thus, dielectric constant is a better predictor of extent of gelatinization of starch slurry than is the loss factor.

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